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# मानक

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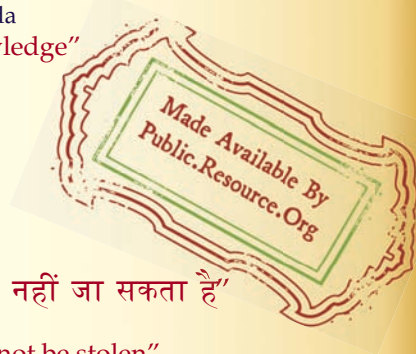
IS 4354-1 (1967): Methods of chemical analysis of magnesium, aluminium brazing alloys, Part 1 Analysis of aluminium, manganese, zinc and silicon [MTD 9: Lead, Zinc, Cadmium, Tin, Antimony and their Alloys]



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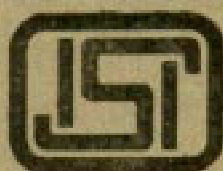
*Indian Standard*

METHODS OF CHEMICAL ANALYSIS OF  
MAGNESIUM-ALUMINIUM BRAZING ALLOYS

PART I ANALYSIS OF ALUMINIUM, MANGANESE,  
ZINC AND SILICON

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*Indian Standard***METHODS OF CHEMICAL ANALYSIS OF  
MAGNESIUM-ALUMINIUM BRAZING ALLOYS****PART I ANALYSIS OF ALUMINIUM, MANGANESE,  
ZINC AND SILICON****Methods of Chemical Analysis Sectional Committee, SMDC 2***Chairman*

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## *Indian Standard*

# METHODS OF CHEMICAL ANALYSIS OF MAGNESIUM-ALUMINIUM BRAZING ALLOYS

## PART I ANALYSIS OF ALUMINIUM, MANGANESE, ZINC AND SILICON

### 0. FOREWORD

**0.1** This Indian Standard was adopted by the Indian Standards Institution on 15 December 1967, after the draft finalized by the Methods of Chemical Analysis Sectional Committee had been approved by the Structural and Metals Division Council.

**0.2** Magnesium-aluminium brazing alloys, the composition of which has been specified in IS : 2927-1964\*, are used for the purpose of joining magnesium-based metals. The purpose of this standard is to prescribe methods for analysis of aluminium, manganese, zinc and silicon. Methods for determination of copper and nickel, which have been restricted to very low amounts in IS : 2927-1964\*, are being investigated and will be covered later in Part II of this standard.

**0.3** In the formulation of this standard due weightage has been given to international co-ordination among the standards and practices prevailing in different countries in addition to relating it to the practices in the field in this country. This has been met by deriving assistance from the '1964 Book of ASTM Methods for chemical analysis of metals: Part 32' issued by the American Society for Testing and Materials.

**0.4** In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960†.

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### 1. SCOPE

**1.1** This standard prescribes methods for determination of aluminium, manganese, zinc and silicon in magnesium-aluminium brazing alloys as specified in IS : 2927-1964\*.

\*Specification for brazing alloys.

†Rules for rounding off numerical values (*revised*).

## 2. QUALITY OF REAGENTS

**2.1** Unless specified otherwise, pure chemicals and distilled water ( see IS : 1070-1960\* ) shall be employed in the tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

## 3. GENERAL

**3.1 Use of Filter Papers** — In some of the methods prescribed in this standard relative numbers of Whatman filter papers, which are commonly used, have been specified. However, any other suitable brand of filter papers of corresponding porosity may also be used.

## 4. DETERMINATION OF SILICON BY THE GRAVIMETRIC METHOD

**4.1 Outline of the Method** — The sample is dissolved in nitric acid and the hydrated silica dehydrated and rendered insoluble by fuming with sulphuric acid. The silica is ignited and determined by the loss in weight after volatilization with hydrofluoric acid in presence of sulphuric acid.

### 4.2 Reagents

**4.2.1 Concentrated Nitric Acid** — sp gr 1.42 (conforming to IS : 264-1950†).

**4.2.2 Concentrated Sulphuric Acid** — sp gr 1.84 (conforming to IS : 266-1961‡).

**4.2.3 Dilute Sulphuric Acid** — 1 : 1 (v/v).

**4.2.4 Hydrofluoric Acid** — 40 percent.

### 4.3 Procedure

**4.3.1** Weigh accurately 5 g of the sample in a covered 400-ml beaker containing 100 ml water and to this add slowly concentrated nitric acid until the sample is dissolved. When the violent reaction has ceased, heat till the solution is complete. Cool, add 12 to 15 ml of concentrated sulphuric acid and evaporate till the nitrous fumes are expelled and white fumes of sulphur trioxide are evolved. Fume for two

\*Specification for water, distilled quality (revised).

†Specification for nitric acid.

‡Specification for sulphuric acid (revised).



to three minutes, cool, and add 200 ml of water. Boil for several minutes till the salts have gone into the solution.

**4.3.2** Filter through filter paper No. 40 containing a little filter pulp of the same quality; using a rubber-tipped glass rod, detach any silica adhering to the beaker. Wash the filter thoroughly with hot water 4 to 5 times.

**4.3.3** Transfer the residue to a platinum crucible. Heat between 500° to 600°C until the filter is completely incinerated, then partly cover the crucible with a platinum cover and ignite in a muffle at 1050°C to constant weight. Allow to cool in a desiccator and weigh. Then moisten the ignited silica with a few drops of dilute sulphuric acid, add 5 ml of hydrofluoric acid and evaporate to dryness. Continue heating until the sulphuric acid fumes are completely eliminated. Complete the ignition in a muffle at 1050°C for 10 to 15 minutes. Allow to cool in a desiccator and weigh. The loss in weight represents silica.

#### 4.4 Calculation

$$\text{Silicon, percent} = \frac{A \times 46.72}{B}$$

where

$A$  = weight in g of the silica obtained from the sample,  
and

$B$  = weight in g of the sample taken.

### 5. DETERMINATION OF ALUMINIUM BY THE OXINE ( GRAVIMETRIC ) METHOD

**5.1 Outline of the Method** — Aluminium is precipitated from the solution as benzoate. The precipitate is dissolved and aluminium precipitated as aluminium oxyquinolate. It is filtered, dried and weighed.

#### 5.2 Reagents

**5.2.1 Dilute Hydrochloric Acid** — 20 percent (  $v/v$  ).

**5.2.2 Bromophenol Blue Alcoholic Solution** — Dissolve 0.20 g of bromophenol blue in 100 ml of ethanol ( 95 percent ).

**5.2.3 Dilute Ammonium Hydroxide Solution** — Dilute 25 ml of concentrated ammonium hydroxide ( sp gr 0.90 ) with water and make up the volume to 100 ml.

**5.2.4 Hydroxylamine Hydrochloride Solution** — Dissolve in a little water 50 g of the reagent (  $\text{NH}_2\text{OH} \cdot \text{HCl}$  ), 50 g of ammonium chloride (  $\text{NH}_4\text{Cl}$  ), 50 ml of glacial acetic acid ( approximately 17.4 N ), and then dilute to 1 000 ml with water.

**5.2.5 Ammonium Benzoate Solution ( 100 g/l )** — Dissolve 100 g of ammonium benzoate in one litre of warm water. It shall be freshly prepared.

**5.2.6 Ammonium Benzoate Wash Solution** — To 100 ml of the ammonium benzoate solution ( *see* 5.2.5 ), add 900 ml of warm water and 20 ml of glacial acetic acid.

**5.2.7 Tartaric Acid Solution** — Mix 500 ml of dilute hydrochloric acid ( *see* 5.2.8 ) with 10 ml of tartaric acid solution ( 500 g/l ).

**5.2.8 Dilute Hydrochloric Acid** — Dilute 25 ml of concentrated hydrochloric acid ( sp gr 1·16 ) with water and make up the volume to 100 ml.

**5.2.9 Sodium Sulphite** — solid.

**5.2.10 Neutral Red Solution** — Dissolve 0·05 g of neutral red in 100 ml of ethanol ( 95 percent ).

**5.2.11 Concentrated Ammonium Hydroxide** — 20 percent.

**5.2.12 Acetic Acid** — Dilute 100 ml of glacial acetic acid ( approx 17·4 N ) with water and make up the volume to one litre.

**5.2.13 8-Hydroxyquinoline Solution ( 50 g/l )** — Dissolve 50 g of 8-hydroxyquinoline in 120 ml of glacial acetic acid and dilute to one litre.

**5.2.14 Ammonium Acetate Solution** — 600 g/l ( w/v ).

### 5.3 Procedure

**5.3.1** Dissolve carefully 0·3 to 0·5 g of the sample in 400-ml beaker in 15 ml of dilute hydrochloric acid. If there is violent reaction, cool the beaker in running water. When the dissolution is complete, dilute to 100 ml if there is copper, filter through paper pulp. Wash the pulp with hot water until free from magnesium.

**5.3.2** Add two or three drops of bromophenol blue solution and neutralize with dilute ammonium hydroxide solution until the indicator becomes violet. Then add 20 ml of hydroxylamine hydrochloride solution and 20 ml of ammonium benzoate solution. Heat the solution to boiling with stirring and keep boiling gently for 5 minutes, then filter through a medium texture filter. Wash the beaker and the precipitate 8 to 10 times with boiling ammonium benzoate wash solution. Discard the filtrate.

**5.3.3** Dissolve the precipitate on the filter with small portions of boiling tartaric acid solution. Wash the filter with warm water and collect the solution and the washings in the first beaker. Transfer the solution to a beaker, add 1 g of sodium sulphite and a few drops of neutral red solution and carefully add concentrated ammonium hydroxide until the indicator

becomes yellow. Dilute the solution to about 200 ml, then heat to about 70°C. Add acetic acid until the indicator becomes red. Add 30 ml of 8-hydroxyquinoline solution and 40 ml of ammonium acetate solution. Allow the precipitate to settle at about 70°C for 30 minutes.

**5.3.4** Filter the precipitate through a tared sintered glass crucible of porosity between 3 and 15 microns ( $\mu\text{m}$ ) by applying weak suction, then wash 6 to 8 times with hot water. Dry to constant weight in an oven at 130°C. Weigh after cooling in a desiccator.

## 5.4 Calculation

$$\text{Aluminium, percent} = \frac{A \times 5.87}{B}$$

where

$A$  = weight in g of aluminium oxyquinolate, and

$B$  = weight in g of the sample taken.

## 6. DETERMINATION OF ZINC BY THE EDTA METHOD

**6.1 Outline of the Method** — Zinc is precipitated as zinc sulphide from buffered formate solution by hydrogen sulphide gas. Zinc sulphide is dissolved in dilute sulphuric acid and titrated with standard EDTA solution.

### 6.2 Reagents

**6.2.1 Dilute Hydrochloric Acid** — 1 : 1 ( $v/v$ ).

**6.2.2 Hydrogen Sulphide** — gas.

**6.2.3 Hydrogen Sulphide Wash Solution** — Dilute 10 ml of concentrated hydrochloric acid with one litre of water and saturate with hydrogen sulphide.

**6.2.4 Ammonium Chloride** — solid.

**6.2.5 Tartaric Acid Solution** — 30 percent ( $w/v$ ).

**6.2.6 Methyl Red Indicator Solution** — Dissolve 0.1 g of methyl red as indicator in 3.72 ml of 0.1 000 N sodium hydroxide solution and dilute to 200 ml. Filter, if necessary.

**6.2.7 Concentrated Ammonium Hydroxide** — 20 percent.

**6.2.8 Formate Mixture Solution** — Add 250 g of ammonium sulphate to a mixture of 200 ml of formic acid (sp gr 1.2) and 30 ml of concentrated ammonium hydroxide. Dilute to one litre with water.

**6.2.9 Formate Wash Solution** — Dilute 25 ml of the above solution ( 6.2.8 ) up to 1 000 ml with water and saturate with hydrogen sulphide gas.

**6.2.10 Dilute Sulphuric Acid** — 1 : 1 ( v/v ).

**6.2.11 Sodium Hydroxide Solution** — 200 g/l.

**6.2.12 Buffer Solution** — Dissolve 54 g of ammonium chloride in 300 ml of water, add 350 ml of ammonium hydroxide and dilute to one litre. This solution has a pH of 10.

**6.2.13 Eriochrome Black-T Indicator Solution** — Dissolve 0.4 g of sodium salt in a mixture of 20 ml of ethyl alcohol and 30 ml of tri-ethanolamine and store in a polythene dropping bottle.

**6.2.14 Standard Disodium Ethylenediamine Tetra-Acetate ( EDTA ) Solution ( 0.05 M )** — Dissolve 18.6 g of the salt in 600 ml of hot water. Cool to room temperature, add 0.1 g of magnesium chloride (  $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$  ) and dilute to one litre with water. Standardize the solution as follows:

Transfer to an 250-ml beaker an aliquot of the standard zinc solution ( 6.2.15 ) approximately equal in zinc content to the aliquot of the sample. Continue as directed in 6.3.4. Calculate the equivalent of the EDTA solution in terms of grams of zinc per millilitre of solution.

**6.2.15 Standard Zinc Solution** — Dissolve 4.000 0 g of pure electrolytic zinc in 200 ml of dilute hydrochloric acid ( 1 : 4 ), cool and dilute to one litre in a volumetric flask.

### 6.3 Procedure

**6.3.1** Dissolve one gram of the accurately weighed sample in a 600-ml beaker by slowly adding 20 ml of dilute hydrochloric acid. Heat till the solution is clear. Dilute to 150 ml with water. Pass hydrogen sulphide gas through the warm solution till the precipitation is complete. Filter on filter pulp. Wash the precipitate with hydrogen sulphide wash solution three or four times. Collect the filtrate and the washings in 500-ml beaker. Reject the residue.

**6.3.2** Boil the filtrate for 5 to 10 minutes to free it from hydrogen sulphide gas completely. Add nearly 10 g of ammonium chloride and then 20 ml of tartaric acid solution. Add few drops of methyl red indicator solution and continue adding concentrated ammonium hydroxide until it is just alkaline to the indicator. Add 25 ml of formate mixture solution and dilute with water to make the volume to 250 ml. Pass hydrogen sulphide gas through the warm solution till the precipitation is complete. Digest the solution at 60°C and then at room temperature to allow the precipitate to settle. Filter on filter pulp and wash the precipitate six to seven times with formate wash solution to make it completely free from magnesium.

**6.3.3** Dissolve zinc sulphide precipitate in hot dilute sulphuric acid. Wash the filter pulp thoroughly five to six times with hot water. Collect the solution and the washings in a 250-ml beaker and evaporate to fumes. Dilute to 30 ml.

**6.3.4** Neutralize with sodium hydroxide solution. Add 30 ml of buffer solution and 5 drops of eriochrome black-T indicator solution. Titrate slowly with standard EDTA solution to a bluish green end point.

#### 6.4 Calculation

$$\text{Zinc, percent} = \frac{A \times B}{C} \times 100$$

where

*A* = volume in ml of the standard EDTA solution required for titration of the test solution,

*B* = zinc equivalent in g per ml of the standard EDTA solution, and

*C* = weight in g of the sample taken.

### 7. DETERMINATION OF MANGANESE BY THE PERIODATE (PHOTOMETRIC) METHOD

**7.1 Outline of the Method** — Manganese is oxidized to permanganate in acid solution with potassium periodate and determined photometrically at about 545 millimicrons ( $\mu\text{m}$ ).

#### 7.2 Reagents

**7.2.1 Dilute Sulphuric Acid** — 1 : 4 (*v/v*).

**7.2.2 Concentrated Acid Nitric** — See 4.2.1.

**7.2.3 Potassium Periodate** — solid.

**7.2.4 Standard Manganese Solution** (1 ml = 0.10 mg of Mn) — Take 0.100 g of pure manganese metal in 150-ml beaker and add 10 ml of dilute nitric acid (1 : 1). Heat till the solution is clear and brown fumes are expelled. Cool and dilute to one litre in a volumetric flask.

#### 7.3 Procedure

**7.3.1** Take accurately weighed one gram sample in 250-ml beaker. Add 15 ml of water and 25 ml of dilute sulphuric acid. Heat till the dissolution is complete. Then add 25 ml of concentrated nitric acid and boil to remove any dark residue. If there is any residue, filter through a fine filter paper. Wash the filter paper three or four times with water. Evaporate the filtrate to about 50 ml.

**7.3.2** Cool slightly and carefully add 0.5 g of potassium periodate. Place on a sand bath for 30 to 40 minutes at a temperature of about 98°C, without bringing to boil. Remove from the sand bath. Allow to cool and make up to 100 ml in a volumetric flask.

**7.3.3** Transfer a suitable portion of the solution to a cell and take the photometric reading of the solution using a light filter at 545 millimicrons (  $\mu\text{m}$  ).

**7.3.4 Calibration Curve** — Take a series of standard manganese solutions and the reagent blank. Using the same quantity of reagents carry through all stages as described under 7.3.2 and record the photometric reading of all the standard solutions along with the blank. Draw a calibration curve by plotting the photometric readings of the standard solutions against milligrams of manganese per hundred millilitres of the solution.

**7.4 Calculation** — Convert the photometric readings of the sample to milligrams of manganese by means of the calibration curve and calculate the percentage of manganese as follows:

$$\text{Manganese, percent} = \frac{A}{B} \times \frac{1}{10}$$

where

$A$  = weight in mg of manganese found in the aliquot of the solution, and

$B$  = weight in g of sample represented by aliquot of the solution taken.

# INTERNATIONAL SYSTEM OF UNITS ( SI UNITS)

## Base Units

QUANTITY	UNIT	SYMBOL
Length	metre	m
Mass	kilogram	kg
Time	second	s
Electric current	ampere	A
Thermodynamic temperature	kelvin	K
Luminous intensity	candela	cd
Amount of substance	mole	mol

## Supplementary Units

QUANTITY	UNIT	SYMBOL
Plane angle	radian	rad
Solid angle	steradian	sr

## Derived Units

QUANTITY	UNIT	SYMBOL	DEFINITION
Force	newton	N	1 N = 1 kg.m/s <sup>2</sup>
Energy	joule	J	1 J = 1 N.m
Power	watt	W	1 W = 1 J/s
Flux	weber	Wb	1 Wb = 1 V.s
Flux density	tesla	T	1 T = 1 Wb/m <sup>2</sup>
Frequency	hertz	Hz	1 Hz = 1 c/s (s <sup>-1</sup> )
Electric conductance	siemens	S	1 S = 1 A/V
Electromotive force	volt	V	1 V = 1 W/A
Pressure, stress	pascal	Pa	1 Pa = 1 N/m <sup>2</sup>

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